

Light coupling and enhanced backscattering in layered plasmonic nanocomposites

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Abstract: Peculiar enhanced backscattering of light as well as selective vapor sensing were recently observed in a layered plasmonic nanocomposite which consisted of gold nanospheres randomly distributed in a sol-gel glass thin film on top of a soda-lime glass substrate, including a buried leaky waveguide. In order to understand the underlying physical mechanisms, we performed three-dimensional transfer-matrix numerical simulations and calculated the reflectance in both backward and specular directions as functions of the incidence angle. First, assuming a layered periodic particle arrangement, we confirmed that backscattering took place at grazing incidence if the spatial period in the layers was chosen within an optimal range, in agreement with theoretical predictions. Then, using a pseudo-random particle arrangement to describe the actual nanocomposite, we revealed that strong backscattering could nevertheless persist for specific particle distributions, in spite of their randomness. This behavior was tentatively explained by putting backscattering in relation with the particle interdistance statistics. Finally, we showed that backscattered reflectance was much more sensitive than specular reflectance to the adsorption of water vapor either on the surface or inside the likely porous structure of the glass host.

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1. Introduction

Plasmonic nanostructures with enhanced and controllable optical properties are key elements in nanophotonics and biosensing [1,2]. In this context, various plasmonic sensing schemes have been developed during the last decades. Simplest schemes are based on adsorbate-induced local refractive index changes at the metallic surface. Current sensors based on metallic films or nanoparticles are able to detect zeptomol concentration variations [3–5]. Aging is an issue for these sensors due to oxidation and solvent-induced nanoparticle reshaping effects [6]. A solution to this problem consists in coating metallic nanostructures with glassy layers. By carefully choosing the layer thickness, it is possible to detect adsorption on the glass surface through changes in the surface plasmons [7,8]. Metal-glass nanocomposites, in which metallic nanoparticles are embedded in a glass matrix [9], are clearly an attractive low-cost alternative solution. In such *layered* plasmonic nanocomposites, one must take into account the facts that plasmonic particles do not lie at the surface and are randomly distributed within a layer. Therefore, novel methods are needed to efficiently excite localized surface plasmons in the embedded particles and to help them to interact with the surrounding medium.

Enhanced backscattering is the result of constructive interference of multiple scattered waves propagating in a medium along common direct and reverse trajectories [10,11]. Enhanced backscattering from embedded dielectric nanoparticles in a metallic slab was predicted numerically [12]. The mechanism involves scattering of the incident light by embedded particles which mediates in-coupling of the light into surface plasmons, and then scattering of the propagating surface plasmons by these particles, resulting in out-coupling of the light. More fundamentally, weak localization of surface plasmons is known to take place at random surfaces [13]. In ref [12], however, the same phenomenon was predicted to occur thanks to multiple scattering of plasmons by embedded particles, in analogy with weak light localization in particle clouds [14].

Recently, enhanced backscattering of light from gold nanospheres embedded in glass (filling fraction of a few percents) was observed in thermally poled nanocomposite glass [15]. In order to enable in-coupling of light into the nanocomposite layer from the incident medium (here the glass substrate), a leaky optical waveguide was intentionally created in the substrate by thermal poling. As a result, the sample was a monolithic analogue to the double-layer Kretschmann geometry used to excite propagating surface plasmons on a metallic film [15]. Light coupling into the leaky waveguide mode was associated with the onset of strong light scattering (1% of incident power) with a significant enhancement in the backward direction

which was not expected in the Rayleigh scattering regime [15]. Interestingly, the backscattered intensity was found to be highly sensitive to volatile organic vapors, with opposite (decrease versus increase) signal evolution for hydrating and dehydrating vapors [15].

In this work, we theoretically study the coupling and enhanced backscattering of light in layered plasmonic nanocomposites, such as the thermally poled sample used in the experiments described above. Using a dedicated three-dimensional (3D) transfer-matrix computer code, we calculate the reflectance of the layered medium in both backward and specular directions, as functions of the incidence angle. In a first step, assuming a layered periodic nanoparticle arrangement, we search for an optimal spatial period which maximizes the backscattering intensity at grazing incidence. Then, taking a pseudo-random particle arrangement to describe the actual sample, we investigate the possibility that enhanced backscattering persists in the presence of randomness. Finally, we evaluate the sensitivity of backscattering to the presence of adsorbed water either on the surface or inside the likely porous structure of the glass host.

2. Sample and models

The sample used in previous experiments was a thermally poled gold/glass nanocomposite consisting of a 130-nm thick sol-gel silica film with randomly dispersed spheroid gold particles (~15 nm diameter, ~2.3% volume filling fraction) on top of a thick soda-lime glass substrate [15]. Within the substrate, just below the film, a 2- μ m thick ion-depleted layer (induced by poling) acted as a buried leaky waveguide layer with a refractive index slightly lower ($\delta n \sim 1\%$) than in the unmodified substrate region (Fig. 1-a). In the experiments, the sample was probed with polarized green laser light (532 nm) from the substrate side, at grazing incidence (with respect to the substrate/film interface) and both backscattered and reflected (specular) light intensities were recorded as a function of the angle, which was tuned around leaky waveguide resonances [15].

For backscattering to take place, regularity of the spatial arrangement of scatterers (here gold particles) was thought to be important. The first theoretical model under investigation (Fig. 1-b) was therefore built by stacking layers of periodic arrays of particles (diameter $b = 15$ nm). A square array (period a) was chosen arbitrarily for all layers. In the stack, layers were alternately shifted by half a period ($a/2$) in both lateral directions (Fig. 1-e) in order to produce a 3-D particle arrangement which was not too far from random arrangement in the sample. The particles were embedded into a SiO_2 host (refractive index $n_{\text{host}} = 1.440$). We had to use up to eight Au/ SiO_2 layers sandwiched by two very thin SiO_2 layers (thickness $h = 5$ nm) in order to obtain a total thickness equal to the actual film thickness ($d = 130$ nm). Between the nanocomposite film and the glass substrate ($n_{\text{sub}} = 1.488$), a reduced-index layer was inserted whose thickness ($d_{\text{depl}} = 2$ μ m) and refractive index (written as $n_{\text{depl}} = (1 - \delta n) \times n_{\text{sub}}$, with $\delta n = 1\%$) were chosen based on the state-of-the-art knowledge of poling [15]. The volume filling fraction of gold in the film was given by:

$$f_{\text{periodic}} = \frac{4\pi b^3}{3a^2 d}. \quad (1)$$

In the sample, particles were randomly dispersed into the film. In order to take into account this aspect and to study its effect on the backscattering, we devised a second theoretical model (Fig. 1-c) where a slab was divided into cubic boxes of size equal to the particle diameter (Fig. 1-f). The slab thickness was taken equal to $8 \times b$ and its lateral dimensions were taken equal to $a = m \times b$ (m integer). As in the periodic model, two thin layers of SiO_2 were added to obtain the actual sample thickness value. Then, N particles were placed randomly into the boxes. The volume filling fraction of gold in the film was given by:

$$f_{\text{random}} = N \frac{\pi b^3}{6a^2 d} = N \frac{\pi b}{6m^2 d}. \quad (2)$$

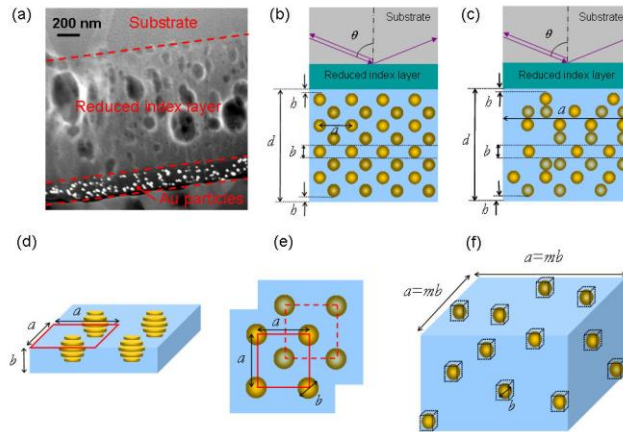


Fig. 1. Plasmonic nanocomposite sample and models. (a) Transmission electron microscopy image of the sample cross-section showing Au particles in a thin sol-gel film on top of a glass substrate whose refractive index was reduced beneath the film as a result of poling-induced ion depletion and formation of void-like nanostructures. (b) Periodic nanocomposite model (cross-sectional view). (c) Pseudo-random nanocomposite model (cross-sectional view). (d) Approximation of spherical Au particles by stacks of five cylinders for numerical computation purposes. (e) In the periodic model (top view), square array layers are alternately shifted by half a period in both lateral directions. (f) In the pseudo-random model, particles are taken randomly in a super-cell box whose dimensions are integer multiples of the particle diameter.

3. Computational method

Three-dimensional transfer-matrix (3D-TM) electromagnetic computational method [16,17] was used to calculate the light intensity that was scattered by the nanocomposite models. The method exactly solves Maxwell's equations for optical media which are arbitrarily stratified (in z direction, normal to layer interfaces) and laterally periodic (in x, y directions, parallel to layer interfaces). The stratified medium concept, which may be applied to both periodic and pseudo-random models of Fig. 1, requires to approximate spherical particles by stacks of cylinders (Fig. 1-d). The 3D-TM method employs spatial Fourier expansion of the dielectric function $\epsilon(x,y)$ for each layer of the structure. By virtue of Bloch's theorem, the electric and magnetic fields are expanded in the same Fourier basis and their Fourier components are propagated throughout the structure by applying electromagnetic boundary conditions at layer interfaces (this calculation is based on Pendry's scattering matrix formalism [16] and leads to both radiative and evanescent field components). The flux of the Poynting vector is calculated in incidence (emergence) medium from field components and the reflectance (transmittance) is deduced for each diffraction direction associated with the lateral periodicity of the structure. In the present study, we retain the reflectance components in backward and specular directions.

The number of plane waves which was needed in Fourier expansion to achieve numerical convergence depended on the topology of the unit cell (number of clusters and their relative position) and on the dielectric constants of the host and cluster materials. For noble metal clusters embedded in glass, the strong refractive index mismatch between clusters and host at visible wavelengths (including imaginary part of the metal refractive index) required a large number of plane waves to achieve convergence [18]. In this work, we used up to 16×16 plane waves (periodic model) and 20×20 plane waves (pseudo-random model) to get numerical results close to convergence within reasonable computation times.

In all simulations, the plane of incidence (see Fig. 1-c,b) was chosen to be aligned along $\hat{e}_x$ direction of the square unit cell. The *internal* incidence angle θ (inside the substrate) was

varied in a narrow range, close to grazing incidence and the wavelength of light was equal to $\lambda = 532$ nm, as in experiments. Incident light polarization was taken to be transverse electric (TE). The complex refractive index of gold was equal to $n_{Au} = 0.467 + i2.407$ at $\lambda = 532$ nm. For computation purpose, each spherical nanoparticle was approximated by a stack of five 3-nm thick cylinders in order to form a compact spheroidal object (Fig. 1-d). We checked that this geometrical approximation had negligible influence on computation results.

4. Simulations results

4.1 Periodic model

Backscattering required that the wavevectors of the incident and elastically scattered waves were equal in magnitude and opposite in direction: i.e. $\mathbf{k}_S = -\mathbf{k}_{inc}$. Since scattering arises from a periodic structure (period a equal to the length of square unit cells in Fig. 1), we applied, as a crude approximation, the grating phase matching condition, i.e. $\mathbf{k}_{S||} = \mathbf{k}_g + \mathbf{k}_{inc||}$, to a planar square array of point scatterers in order to predict the optimal period for backscattering ($\mathbf{k}_g$: grating vector, $k_{inc||} = 2\pi/\lambda \times n_{sub} \times \sin\theta$: component of incident wavevector parallel to the grating plane). For the light that was incident in the (x,z) plane at grazing incidence, phase matching for the diffraction order that scattered light backwards imposed $k_g = 2 \times k_{inc||}$ and the period was therefore given by: $a = \lambda/(2 \times n_{sub} \times \sin\theta)$. At grazing incidence ($\theta \sim \pi/2$), the optimal period was estimated to be $a \sim 180$ nm using $\lambda = 532$ nm and $n_{sub} = 1.488$.

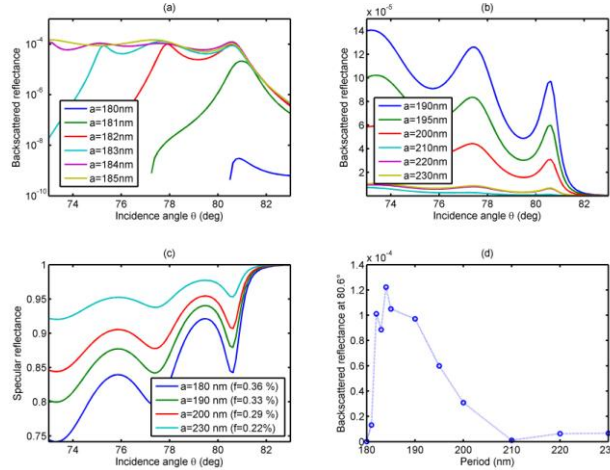


Fig. 2. Reflectance of periodic gold nanoparticle arrangements with periods varying from $a = 180$ nm to 230 nm. (a) Backscattered reflectance angular spectra (logarithmic scale) for periods ranging from $a = 180$ nm to 185 nm. (b) Backscattered reflectance angular spectra for periods ranging from $a = 190$ nm to 230 nm. (c) Specular reflectance angular spectra for periods ranging from $a = 180$ nm to 230 nm (gold filling factor f calculated by Eq. (1) is indicated between parentheses). (d) Backscattered reflectance at resonance angle $\theta = 80.6^\circ$ as function of the period.

Simulations were performed on the periodic model with different values of the period. We first checked the occurrence of leaky waveguide resonances at grazing incidence by calculating the angular spectrum of the specular reflectance component (Fig. 2-c). Resonances were found at 80.6° , 77.4° , 73.2° whose depth increased with increasing gold filling fraction, see Eq. (1). Same conclusions were obtained using effective medium approximation of gold/glass nanocomposite layers. We then calculated the reflectance component related to the backward diffraction direction and found that backscattering showed up for periods higher than $a \sim 180$ nm (in agreement with the predicted value), developing peaks around the angles associated with leaky waveguide resonances (Fig. 2-a,b). This result confirmed that the existence of a reduced-index layer was mandatory to enable interaction of the incident light

with the gold particle surface plasmons in general, and to produce backscattering in particular. As the period was increased from 180 nm, the backscattering intensity at resonance angle $\theta = 80.6^\circ$ increased sharply up to a maximum of $\sim 1.2 \times 10^{-4}$, for $a = 184$ nm, and then decreased slowly (Fig. 2-d). We also checked that electromagnetic waves associated with all other diffraction directions were evanescent and therefore gave no contribution to the reflectance.

4.2 Pseudo-random model

In the frame of the periodic model, the gold volume filling fraction, as predicted by Eq. (1), was found to be one order of magnitude lower than in the sample ($\sim 0.3\%$ instead of $\sim 2.3\%$, see Fig. 2). This large mismatch together with the random character of the particle distribution in our sample (Fig. 1-a) led us to devise a pseudo-random model. In this model, the nanocomposite film was built from a rectangular cuboid super-cell which was divided into $m \times m \times 8$ cubic boxes with the gold particles randomly distributed within them (Fig. 1-f). Note that, because the particle positions were randomly distributed *in the whole volume* of the super-cell, the number of particles could be different in each of the eight layers (Fig. 1-c). The idea behind the pseudo-random model was to take a large super-cell and fill all the layers with a suitable number of randomly distributed particles matching the volume gold filling fraction in the sample. The point was therefore to investigate the possibility that particle interdistances could be *statistically* close to the optimal period for backscattering. The pseudo-random nature of the model came from the fact that the length of the super cell introduced an *artificial* period in the structure. Ideally, in order to avoid statistical bias, the super-cell length should be much larger than the expected *physical* period (§ 4.1). However, due to practical limitations in the computation time, we had to restrict the number of gold particles and to take a super-cell length equal to $a = 210$ nm ($= m \times b$, with $m = 14$). By taking $N = 75$ particles in the super-cell box (Fig. 1-f), the gold volume filling fraction, as predicted now by Eq. (2), was equal to 2.31%, i.e. very close to the experimental value ($\sim 2.3\%$).

The pseudo-random model was tested with 12 statistical realizations. The gold volume filling fraction being constant for all realizations, the specular reflectance was not much influenced by the particle distribution (Fig. 3-a) as it could be anticipated from effective medium considerations (variations in reflectance were likely to be due to different particle concentration gradients across the depth of the film). By contrast, the backscattered reflectance changed by one order of magnitude depending on the particle distribution (Fig. 3-b). The origin of this effect was investigated further by assuming that particles being closest to the substrate (first nanocomposite layer from the top in Fig. 1-c) predominantly determined the backscattering characteristics. We had an indication that it was indeed the case from simulations of the periodic model using one and eight layers, respectively: although leaky waveguide resonances were strongly reduced in the specular reflectance spectrum when taking only one layer instead of eight (thickness of gold/glass nanocomposite film reduced accordingly), backscattered reflectance peaks had similar levels in both cases (this was attributed to the shift of the particle array from one layer to the next in the periodic model which avoided constructive interference between arrays).

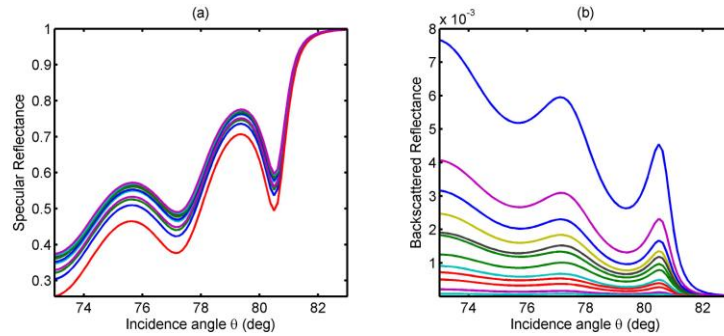


Fig. 3. Reflectance of 12 statistical realizations of pseudo-random gold nanoparticle arrangements (gold volume filling factor f calculated by Eq. (2) is 2.3%). (a) Specular reflectance angular spectra. (b) Backscattered reflectance angular spectra.

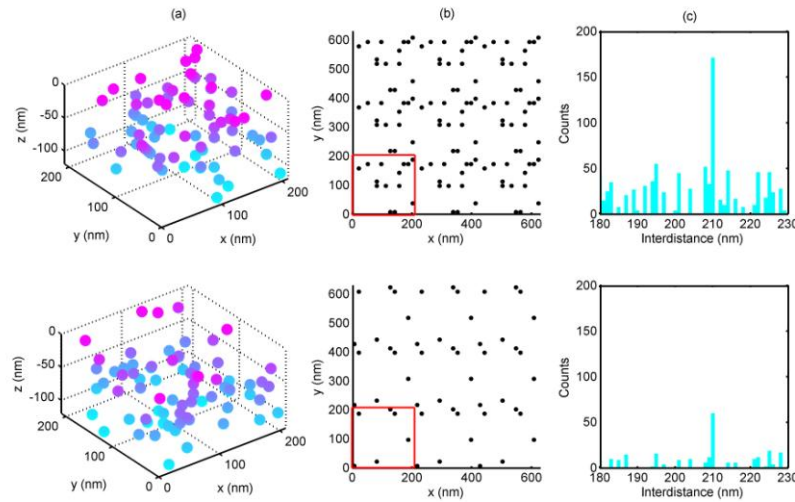


Fig. 4. Statistics of particle distribution for pseudo random gold nanoparticle arrangements leading to highest (upper charts) and lowest (lower charts) backscattering according to Fig. 3. (a) 3D particle distributions in the super-cell of the film. (b) 2D particle distributions in 9 super-cells of the first layer of particles (super-cell highlighted in red). (c) Histograms of the 2D particle interdistance computed from (b).

In order to understand the effect of disorder in particle positions, we selected the particle distributions that led to highest and lowest backscattering, respectively (Fig. 4-a). Statistics of the particle distribution within the first layer (Fig. 4-b) were investigated and histograms of the particle interdistance were computed from 9 super-cells (Fig. 4-c). First of all, we noted that the model with highest backscattering had a higher number of particles in the first layer super-cell (14 instead of 6 particles). Secondly, the particle interdistance in this case had more counts in the range corresponding to the optimal period of the periodic model (180-200 nm, cf. Fig. 2-d). These results supported our previous experimental observations that random plasmonic nanocomposites could exhibit enhanced backscattering, provided that the particle interdistance was *on average* close to the optimal period set by the phase matching condition [15]. Actually, TEM images of the sample cross section gave indication of particle clustering with average distance of about 200 nm (Fig. 1-a). Actually, similar behavior was observed in colloidal liquids where short-range-order induced backscattering resonances [19,20].

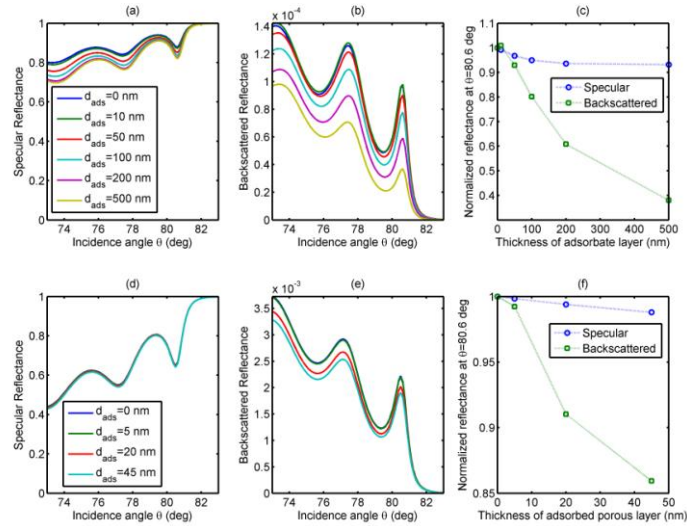


Fig. 5. Sensitivity of plasmonic nanocomposite models to water adsorption on external surface (top graphs, periodic model, d_{ads} : thickness of water layer) or inside pores of host glass (bottom graphs, pseudo-random model giving highest backscattering, d_{ads} : thickness of porous nanocomposite layer). (a,d) Specular reflectance spectra. (b,e) Backscattered reflectance spectra. (c,f) Backscattered reflectance at resonance angle $\theta = 80.6^\circ$ as function of d_{ads} .

6. Sensing

Previously reported experiments showed a rapid change of the detected backscattering signal, followed by saturation, when an open recipient with hydrating (dehydrating) vapors, or even a finger (moisture), was approached from the external film surface [15]. The origin of this spectacular sensing effect (finger-tuned plasmonic response!) was speculated to arise from modifications of the dielectric environment surrounding the plasmonic particles located near the surface. In photonic sensors, adsorption of vapors can take place either on the active surface [4,21] or inside its porous structure [22,23]. In order to test the first sensing scheme, we studied the influence on the reflectance of water which could be adsorbed on the surface, forming thin homogeneous layers of refractive index $n = 1.33$. Both the specular reflectance (Fig. 5-a) and the backscattered reflectance (Fig. 5-b) of the periodic model ($a = 190 \text{ nm}$) were found to decrease with increasing thickness of water layer but the latter was much more sensitive than the former (Fig. 5-c). The decrease of backscattering with adsorbed layer thickness was consistent with the observed decay of the sensing signal as hydrating vapors were adsorbed on the sample [15]. In order to test the second sensing scheme, we speculated that the sol-gel host glass could be porous and water from ambient humidity could be infiltrated within the pores of the nanocomposite layers close to the surface. The aim was to understand the opposite sensing behaviors observed with hydrating/dehydrating vapors. In order to model the *porous* nanocomposite, we assumed that the three bottom layers of the stack (one layer of thickness h , two layers of thickness b) were soaked with water and we treated the host glass as effective glass/water medium (water filling fraction $f_{pore} = 30\%$). We calculated then the specular and backscattered reflectance of the most sensitive pseudo-random model for the dry and partially wet structures (Fig. 5-d,e). Indeed, backscattering was found to be more sensitive than specular reflectance to the dielectric environment of the plasmonic particles as it became modified by the presence of water in the glass pores (Fig. 5-f). According to this model, dehydrating (hydrating) vapors were expected to increase (decrease) the signal, as observed in experiments.

7. Conclusion

Enhanced interaction of light with localized surface plasmons in embedded gold nanoparticles was studied theoretically. Leaky-waveguide-type coupling of light into the plasmonic nanocomposite film at grazing incidence enabled to obtain light reflection in both specular and backward directions. Numerical 3D-TM simulations of the plasmonic nanocomposite reflectance revealed that enhanced backscattering could be obtained from random particle arrangement provided that particle interdistance statistics was appropriate. The high sensitivity of backscattering to vapor adsorption at the nanometer scale, with discrimination between hydrating and dehydrating vapors, is believed to be useful for selective vapor sensing. In practice, the backward light detection scheme can be implemented with a bidirectional optical fiber, making the sensor robust to misalignment as opposed to a specular light detection scheme.

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